MAGNETIC STUDY OF IRON-CONTAINING GLASSES AT ROOM TEMPERATURE

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ABSTRACT. The well-known magnetic method of analysis is applied to elucidate the origin of colour in boric oxide, borate and high lead silicate glasses containing iron. It is observed that iron exists in glass in more than one form and the proportion of different forms of iron in glass changes with the (1) composition of the glass base, (2) the nature and concentration of iron compound and (3) the temperature and the time of heating. All these observations are discussed with reference to the existing theories about the origin of colour in iron glass. The above observations are also explained from the structural picture of each glass base. The colour of iron glass comprising solid phase colours and liquid phase colours is due to the resultant effect of two or more forms of iron of different valency states with the different types of electronic binding including the covalent and electrovalent linkages.

INTRODUCTION

The study of the origin of colour in iron glass has provided a fascinating problem from the early time onwards. Outstanding workers on this line are Jackson, Weyl, Moore, Jung, Stevels, Cole, Kreidl and few others. Though the published information is vast and voluminous yet our knowledge of the mechanism of coloration in iron glass is still elementary. An excellent summary of the different schools of thought has been compiled by Weyl (1950) in a monograph on coloured glasses. But from the results of several workers like Moore *et al* (1949 and 1950), Weyl (1943), Cole (1951*a*) Stevels (1950), Jong (1952, 1954) and Abou-El-Azm (1954), it is clear that there is no consensus of opinion regarding the chemical picture of different chromophores in iron glass. Their observations are summarised in Table I.

Perhaps the most distinctive property of different forms of iron is their magnetic behaviour. The nature of bondings of iron ion with its surrounding ions, its co-ordination number, valency etc. have a direct influence on the magnetic property of the element. Further it is known from the work of Cole (1951*b*) that the bondings with oxygen in the network forming position are more covalent than the bondings in the network modifying position. Naturally with the transition of the electronic bonding to the more covalent type of bonding the paramagnetic susceptibility will decrease by the quenching of the orbital

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moments. In the Table II, the susceptibility values of iron are given in various states of valency and coordination which will be useful for comparison with our experimental findings.

As such, it will be interesting to study by magnetic method the state of existence of iron in glasses of varying compositions as well as in the specimens prepared under different melting conditions. There, too, the effect of concentration of colourant and the relationship between the colour and the state of existence of iron in 'neutral' *glasses and in some 'reduced' †glasses as well as in a few 'oxidised' glasses deserve careful attention

TABLE 1

Description of colour	Name of different workers with their views given in the vertical column				
	Moore and Co-workers (1949, 1951 & 1951)	W.A.Weyl (1943)	H.Cole (1951)	J.M Stevels (1950)	J.De Jong (1952 & 1954)
Colourless (Absorption in ultraviolet)	FeO_4 Tetrahedra	Fe^{+3} N W.M.*		Fe^{+2} N W.M.	Fe^{+3} N.W F
Brown	Fe_2O_3 Colloidal	Fe^{+3} N.W.F.*	Fe^{+3}	Fe^{+3} N W.F.	
Blue	Fe^{+2}	Presence of a group $Fe^{+2}-O-Fe^{+3}$	Fe^{+2}	Interaction of $Fe^{+2}-Fe^{+3}$ (probably net. work forming)	
Colourless (Absorption in Infra-red)		Fe^{+2} N.W.M.		Fe^{+2} both N.W.F. and N W.M.	Fe^{+2} N.W.F
Grey	Fe_3O_4 Colloidal			Mixture of Fe^{+3} (N.W.F.) and $Fe^{+2}-Fe^{+3}$	

*Where N.W M. means network modifying cation and N.W.F. means network forming cation

†Glasses are prepared in neutral or oxidising or reducing furnace atmosphere.

TABLE II

Magnetic property of some forms of iron

Description of iron ion with no electron pairing	χ of Fe $\times 10^6$ at 25°C	Description of iron ion with electron pairing	χ of Fe $\times 10^6$ at 25°C	Description of some oxides of iron	Magnetic behaviour
Fe^{+2} with orbital moment	225 4	Fe^{+2} in square configuration	59 68	FeO in solid aggregates	Feebly paramagnetic
Fe^{+2} spin only value	178 8	Fe^{+2} in Octahedron	Dia-magnetic		
Fe^{+3} spin only value	261.1	Fe^{+3} in square	111 6	α-Fe_2O_3 in solid aggregates	Feebly Ferro-magnetic
Fe^{+4} with orbital moment	225 4	Fe^{+3} in Octahedron	22 29	α-Fe^2O_3 in colloidal dispersion in glass	$\chi^* = 39.4 \times 10^{-6}$ at 20°C (after Abou-El-Azm, 1954)
Fe^{+4} without orbital moment	178 8	Fe^{+4} in square	178.8	γ-Fe_2O_3 in solid aggregates	Ferromagnetic.
Fe^{+6} with orbital moment	178.8	Fe^{+4} in Octahedron	59 68		
Fe^{+6} without orbital moment	59 68	Fe^{+6} in square	59 68	Fe_3O_4 in solid aggregate	Ferromagnetic
		Fe^{+6} in Octahedron	59.68	Fe_3O_4 in colloidal dispersion in glass	$\chi^* = 424 \times 10^{-6}$ at 20°C (after Abou-El-Azm, 1954)

*Different values are also found in the literature.

EXPERIMENTAL PROCEDURE

The present investigation covers several glass systems such as primary, binary and ternary iron glass where boric oxide is the main glass forming oxide. Glass series studied are as follows . B_2O_3, alkali-boric oxide and lithia-beryllium oxide boric oxide glass systems. The last named glass is usually known as Lindemann glass. A variety of high lead potash silica glass is also included.

All experimental specimens were prepared from extra pure materials such as borax, boric oxide, alkali carbonate, alkali nitrate, beryllium carbonate, red lead (Pb_3O_4), ferrous exalate, tartaric acid, precipitated silica etc. Boric oxide was prepared from pure boric acid by heating at 1260°C in an electric furnace for eight hours. The requisite proportions of different components of glass batch were thoroughly powdered, mixed and then heated at 1050°C for six hours in a platinum crucible, except in the case of lead glass where sillimanite crucible was used. All glass samples were prepared in a neutral furnace atmosphere with the

exception of few 'reduced' borate glasses and 'oxidised' lead silicate glasses. Special care was taken to prevent frothing in some high alkali glass batch by adding the batch compositions in small quantities to the melting pot till they melt. The melt of silicate glass was agitated several times during the course of melting so as to ensure complete homogeneity of the system. Further all glasses were annealed at 575°C for half an hour and then cooled to room temperature in the same way prior to any physical examination.

The total iron content of each specimen was determined chemically by the usual dichromate process after removing the boric oxide as methyl borate. In the case of lead glass and Lindemann glass (lithia beryllium oxide-boric oxide glass) necessary steps were taken for the removal of interfering elements like lead and silica in lead glass and beryllium in Lindemann glass respectively before determining the iron content in the system. The alkali content in each glass specimen was calculated from batch composition

All magnetic measurements were carried out in an accurate and sensitive torsion type Curie balance which was developed and modified in our laboratory. The details of the balance with the method of calibration and the limit of accuracy have already been published by Dutta-Roy (1955).

The susceptibility at room temperature 300°K is calculated from the standard expression :

$$\chi_1 = \frac{m_2\theta_1}{m_1\theta_2}\left(\chi_2 - \frac{K_a}{P_2}\right) + \frac{K_a}{P_1}$$ where the correction for air has been introduced.

χ_1 = mass susceptibility of the unknown substance at 300°K.
χ_2 = mass susceptibility of the standard substance at 300°K.
m_2 = mass of the standard substance.
m_1 = mass of the unknown substance.
θ_1 = rotation in degrees for the unknown substance of the torsion fibre to balance the magnetic force.
θ_2 = Corresponding rotation in degrees for the standard substance.
K_a = volume susceptibility of air at 300°K.
P_1 = density of the unknown substance at 300°K.
P_2 = density of the known standard substance at 300°K.

The standard substance used in the present investigation was chromium potassium alum. The value for the mean square of the effective moment of Cr^{+3} is 15.05 in Bohr unit [Dutta-Roy, (1955)] . For pure glass base which is diamagnetic, water was taken as a standard substance with mass susceptibility at 300°K $= 0.7200 \times 10^{-6}$. Mean of three measurements of each specimen was taken.

In the vitreous system like iron-glass the magnetic property of iron is affected much by the diamagnetic property of the glass base, due to the small concentration of the colouring constituent. However, the mass susceptibility of iron can be determined from the following additivity relation :

$$\chi_g = \chi_c\, C_r + \chi_b(1-C_r)$$

where χ_g = mass susceptibility of glass
χ_c = mean mass susceptibility of iron
χ_b = mass susceptibility of the glass base
C_r = concentration of iron

Thus the mass susceptibilities obtained for the iron in different types of iron glasses were compared with one another amongst several glass series studied here

MAGNETIC PROPERTY OF IRON IN BORIC OXIDE GLASS

In the literature some valuable observations have been reported on the colour mechanism of borate and borosilicate glasses containing iron by Moore, Weyl, Abd-El-Moneim etc. but so far no such work has been done on pure B_2O_3 series possibly due to the difficulty of preparing boric-oxide iron glass where the colouring oxide Fe_2O_3 (in the pure form) does not satisfy the criteria of glass forming oxide. However, it is observed that iron oxide can be dispersed in pure dry boric oxide at a temperature of 1050°C to the extent of 0.3 per cent iron (of the total glass content) and the resultant colour of the glass is yellow. Two such samples with varying iron content have been studied. It is observed that the colour of the glass deepens more with the increasing content of colouring constituent in glass.

The susceptibility value of iron in each sample is given in the following table :

TABLE III

Boric Oxide Glass

Glass sample No.	Iron content in weight percent	χ of glass $\times 10^6$ at 25°C	χ of glass base $\times 10^6$	χ of iron $\times 10^6$ at 25°C
1	0 1722	−0 2981	−0.4300	73.70
2	0.2712	−0 1983	−0.4300	84.98

It can be readily seen from Table III that the susceptibility of iron in B_2O_3 glass does not correspond with the value of any single form of iron. Again, from the consideration of the glass composition it is evident that Fe_2O_3 in itself or in combination with B_2O_3 cannot form network forming or network modifying

cations. All these indicate that iron exists in two or more forms as solid phase colours in the glassy matrix. The distribution of different forms of iron is very much dependent on the total iron content in the same glass base.

MAGNETIC PROPERTY OF IRON IN ALKALI-BORIC OXIDE SERIES

The present investigation of different binary borate systems is extended to various samples with increasing alkali content in lithia-boric-oxide, soda-boric oxide and potash-boric oxide glasses. The magnetic work on similar glass systems is already reported by Abd-El-Monem (1954) and Bhatnagar (1945).

The experimental specimens comprise a number of samples of different compositions and each glass specimen was prepared in neutral furnace atmosphere. In all these cases, iron was introduced in the original glass base as Fe_2O_3. The mass susceptibility of iron in different glass bases is given in the following table :

TABLE IV

Lithia-boric oxide series

Glass sample No.	Alkali oxide in wt. percent	Fe in weight percent	χ of glass $\times 10^6$ at 25°C	χ of glass base $\times 10^6$	χ of Fe $\times 10^6$ at 25°C
3	5.745	1 745	3 587	−0.3115	223.0
4	10.04	1.672	3 530	−0.4173	235 7
5	15 00	1 013	2.055	- 0 4160	243.5
6	18.483	0 72	1 337	- 0.4193	243 5
7	18.483	1 010	2.099	−0 4193	249 1
8	18 483	1 119	2 411	−0 4193	252.5
9	31 20	0.250	0.2150	−0.3971	244.4

Soda-boric oxide series

10	9 475	2.848	5.534	−0 4213	208.6
11	17 46	1 585	5.271	−0.4432	225 8
12	31 00	0.650	1 116	−0 4217	236 1
13	31.00	1 048	2.137	−0.4217	243.6
14	31 00	1 210	2.571	- 0.4217	246.9
15	34.00	1 358	2.870	−0.3989	240 2

Potash-boric oxide series

16	4.615	1 621	2.629	−0 4580	190 0
17	14 22	1.673	3.120	−0.4186	211.1
18	41 44	0.830	1.556	- 0.4155	237.1
19	41.44	1.221	2.528	−0.4155	240 6
20	41 44	1.310	2 789	−0.4155	244.2
21	58.55	0.421	0.790	−0 4213	243.1

It is evident from Table IV that the mass susceptibility value of iron changes with the change of glass composition, nature and the content of alkali ion. In the same weight for weight composition of different alkali-boric oxide glasses, the mass susceptibility value of iron decreases from lithia-boric oxide glass to potash-boric oxide glass. Further, the experimental susceptibility value of iron does not correspond with the theoretical value of any likely form of iron. Again, in comparison with the mass susceptibility value of iron in B_2O_3 glass, the mass susceptibility value of iron in the present binary system is quite high, which is due to the presence of more of high-mass-susceptibility form of iron like ferric ion in the system. Further, in all cases the mass susceptibility value of iron in binary system lies between the susceptibility value of ferrous and ferric ions.

It may be of interest to mention here that the present result is not in agreement with that of Abou-El-Azm or Bhatnagar. The value of mass susceptibility of iron in the above soda-boric oxide glass as well as in other lithia-boric oxide and potash-boric oxide glasses of composition similar to ours and of similar thermal history as obtained by Abou-El-Asm lies much below the Fe(ous) 'spin only' value of iron in each case. In contrast with that the value of mass susceptibility of iron in 'oxidised' borax (with 31% Na_2O) glass as obtained by Bhatnagar (who studied only few samples of soda-boric oxide glasses) is very close to the ferric 'spin only' value. Whereas our results of mass susceptibility of iron lie in between those of ferous and ferric 'spin only' values.

MAGNETIC PROPERTY OF IRON IN TERNARY BORATE GLASS

The ternary borate system is represented by lithia-beryllium oxide-boric oxide glass (usually called Lindemann glass) of the following percentage composition : $Li_2O = 13.74$, $BeO = 5.54$ and $B_2O_3 = 80.72$ The colouring constituent is introduced in the glass as Fe_2O_3 The choice of the above system has been made after due consideration of the structure of lithia-boric oxide glasses. In connection with the constitution of ternary borate glass, it is worthwhile to mention here that the above system contains small amount of Be^{+2} ion which has a small ionic radius ($r = .31$ Å) and high ionic potential (8.6).

The mass susceptibility of iron in Lindemann glass is given in the following table wherein the susceptibility value of iron in two binary lithia-boric oxide glasses with 10 and 15 per cent lithia content respectively is included for the sake of comparison.

It is seen from Table V that the susceptibility value of iron in Lindemann glass lies in between that of ferrous and ferric ions and this value is greater than the corresponding lithia-boric oxide glass (vide table II). This can be accounted for by the presence of Be^{+2} ion in Lindeman glass where Be^{+2} ion can also function as a network forming cation. Thus in the competition for occupying the network

TABLE V

Lindemann Glass

Glass sample No.	Type of glass	Lithia in wt per cent	Fe in weight per cent	χ of glass $\times 10^6$ at 25°C	χ of glass base $\times 10^6$	χ of Fe $\times 10^6$ at 25°C
22	Lindemann glass	13 74	2 004	4.515	−0.4425	247.0
23	,,	13 74	1.100	2 246	−0.4425	244.0
4	Lithia-boric oxide	10 04	1 672	3.530	−0 4173	235.7
5	Lithia-boric oxide	15 00	1.013	2 055	−0.4160	243.5

forming position in the glass meshwork in both Lindemann glass and lithia-boric oxide glass, the number of ferric ion in network forming position will be usually smaller in lithia-boric oxide glass and still smaller in Lindemann glass. That is why the possibility of the greater proportion of ferric ion in network modifying position is more in Lindemann glass than in the corresponding lithia-boric oxide glass. So the susceptibility value of iron in Lindemann glass is more than the corresponding lithia-boric oxide glass.

MAGNETIC PROPERTY OF IRON IN POTASH-LEAD OXIDE-SILICA 'OXIDISED' GLASS

The potash-lead oxide silica glass of percentage composition, PbO = 60.0, K_2O = 10.0 and SiO_2 = 30.0 stands quite in contrast with other glass systems studied in the present case. The major constituent in this case is lead ion Pb^{+2} with its high polarizability capacity with (18+2) outer electrons In this glass batch Fe_2O_3 was added as colouring oxide and potassium was introduced as KNO_3 so as to ensure oxidising condition during the period of melting. The colour of this glass is yellow and in such high-lead glasses, small amount of iron produces intensive colour [(Jackson, 1927) and Hampton, (1946)]. This type of glass has been described by Moore and co-workers as fully ferric glass where iron exists mainly as a colouring ferric ion. The susceptibility value of iron in high lead silica glass as obtained by us is given in the following table .

TABLE VI

Potash high lead silica glass

Glass sample No.	Iron in weight per cent	X of glass $\times 10^6$ at 25°C	X of glass base $\times 10^6$	X of iron $\times 10^6$ at 25°C
24	0.78	1.614	−0.3259	242.2
25	4.01	10.110	−0.3259	259 9

It is quite evident from Table VI that the susceptibility value of iron in the above glass series with higher iron content shows a close similarity to that of the ferric ion, although in the case of low iron content specimen the mass susceptibility of iron is somewhat lower than the corresponding ferric iron. It is to be noted here that Moore and Kumar (1951) and Abou-El-Azm (1954) studied the similar type of glass and their experimental data are discussed in this connection. Moore and Kumar did also record like us, the gradual increase of mass susceptibility value of iron in glass with the increase of iron content in the glass system but their experimental values stand quite high (χ of iron $= 330 \times 10^{-6}$ at 20°C) compared to those of ours. But Abou-El-Axm's data are too low (χ of iron $= 133.8 \times 10^{-6}$ at 20°C). Again when compared with other glass systems studied here, the susceptibility value of iron in this glass system is usually high, specially in a high iron content specimen (4.01%). Thus the composition and the oxidising condition of the above glass batch favour the formation of the higher mass susceptibility variety of iron like ferric ions in the system.

THE EFFECT OF IRON CONCENTRATION ON THE SUSCEPTIBILITY VALUE OF IRON IN GLASS

It is quite evident from the preceding tables (I to VI) that the mass susceptibility value of iron in the same glass base increases with the rise of iron content. This observation opens a number of speculations about the relationship between the concentration and proportion of different forms of iron including the ferromagnetic form of iron.

In order to identify the ferromagnetic component in the glassy matrix, if there be any, the magnetic measurements of almost all samples of preceding tables were extended to different fields strength (3500 gauss and 2500 gauss) and in each case the same result was obtained. The above observation rules out any possibility of the existence of any discrete ferromagnetic substance in the glassy matrix.

It is to be noted here that the influence of concentration of iron on the colour of iron-glass has been studied by some workers like Che Andresen Kraft (1931), Fuwa (1935-38), Densem and Turner (1938), Wang and Turner (1942), Abou-El-Azm and a few others. Besides the spectro-optical absorption study, Abou-El-Azm studied the relationship between the concentration of iron and the mass susceptibility of iron in the same glass of alkali-silicate series. He observed that with the increase of iron concentration, there is a corresponding increase in the susceptibility value of iron.

THE STATE OF IRON IN GLASS PREPARED UNDER REDUCING CONDITION

Hitherto, all the work has been done in the glass systems wherein iron was introduced in the form of Fe_2O_3 and the colour of the glass was yellow in general.

But a variety of blue or bluish green coloured iron glass can be prepared by adjusting the composition of the glass base and having a reducing atmosphere in the furnace. This condition is nearly fulfilled by using iron-oxalate in place of Fe_2O_3 and the partial replacement of alkali oxide by alkali tartarate or alkali oxide with tartaric acid in the glass batch composition Some typical alkali-boric oxide glasses have been prepared under these conditions and the colour of these glasses is bluish green. Abou-El-Azm (1954) and Bhatnagar (1954) had studied some such borate glasses. It is observed that the reduction of iron in potassium borate is easier than that in sodium borate glass. The mass susceptibility value of iron in 'reduced' sodium diborate and potassium diborate glasses are given in the following table along with the corresponding values of 'yellow' (neutral) glasses with the same iron content

TABLE VII

Glass sample No	Description of glass	Alkali oxide in wt. per cent	Fe in weight per cent	χ of glass $\times 10^6$ at 25°C	χ of glass base $\times 10^6$	χ of Fe $\times 10^6$ at 25°C
26	Soda boric oxide reduced	31 00	1 210	2.389	—0 4217	238 9
14	Soda boric oxide neutral.	31 00	1.210	2 574	--0.4217	246.9
27	Potash-boric oxide reduced	41 40	1.221	2 086	0 4155	204 5
19	Potash-boric oxide neutral	41.40	1 221	2 528	– 0 4155	240 6

It is quite evident from Table VII that the mass susceptibility value of iron in so-called ferrous glass is less than that of the corresponding yellow ferric glass but this observation is not in agreement with the results of Abou-El-Azm who found that the mass susceptibility of Fe in reduced alkali borate glass is more than the corresponding neutral glass Bhatnagar (1945) however observed that the mass susceptibility value of iron in reduced glass was lower than the corresponding oxidised or netural glass That is quite clear from the consideration of the fact that the mass susceptibility value of the ferrous ion is less than that of the ferric ion. Again the overall mass susceptibility value of iron in ferrous glass is greater than the mass susceptibility value of pure ferrous ion and that indicates the presence of ferric ion in the system. As a matter of fact it is almost impossible to have all the iron in divalent state as is evident from the work of Bancroft and Cunningham (1930) and Weyl (1943).

MAGNETIC STUDY OF SOME GLASS SAMPLES OF DIFFERENT THERMAL HISTORY

In the course of the present investigation, the influence of temperature and time of heating on the susceptibility of some iron glasses have been studied.

Some glass specimens have been thermally treated at various temperatures without, of course, devitrifying the glass as a whole, with a view to studying the change in the magnetic property of those samples It is well known that the glass as a class is very much susceptible to temperature treatment. As a matter of fact all its physical and thermodynamic properties are dependent on its thermal history. So in the same iron glass, it is quite expected that the relative proportion of ferrous and ferric state of iron in the glassy matrix will be affected very much due to the varying way of temperature treatment. With the increasing of time and temperature of melting, the colour of the same glass undergoes a change. As, for example, the colour of the ferric glass changes from yellow to grey through some intermediate stages during the process of long heating. Similar is the case with ferrous glass where the colour changes from blue to green and finally to grey through some intermediate states. The change of colour at different colouring stages is associated with the change of different forms of iron already present in the glass. Consequently along with that, the overall susceptibility value of iron in glass changes with the change of colour. Thus the different forms of iron undergo oxidation, reduction as the case may be, with the tempera-

TABLE VIII

Glass sample No	Description of glass*	Alkali oxide in wt per cent	Fe in weight per cent	Thermal history		χ of glass $\times 10^6$ at 25°C	χ of Fe $\times 10^6$ at 25°C
				Temp. in °C	Time in hours		
28	Soda-boric oxide	9 475	2 848	900	3	5.272	199.5
29	–do	9 475	2.848	1050	3	5 525	208.4
10	–do–	9 475	2 848	1050	6	5.534	208 6
30	-do-	9 475	2 848	1200	3	6 120	230.0
31	–do–	9 475	2 848	1350	3	7 509	278 1
32	–do–	31.00	1 048	1050	3	2 005	231 2
13	-do–	31 00	1.048	1050	6	2.137	243 6
33	Potash-boric oxide	41 40	1 221	825	2	2 276	219.7
34	–do–	41 40	1 221	825	4	2.314	222.8
35	–do–	41 40	1 221	1050	1½	2 470	235 6
36	-do–	41.40	1 221	1050	3	2 496	237 8
19	–do–	41.40	1.221	1050	6	2.528	240.6
1	Boric oxide	0	0.172	1050	6	−0.298	73.7
37	–do–	0	0.172	1050	16	−0 052	218 7

*Glasses prepared in neutral furnace atmosphere.

ture of melting, duration of heating and the furnace atmosphere. Ferrous glass in the absence of reducing furnace atmosphere gets oxidised to ferric state on prolonged heating. In ferric glass there is a possibility of formation of a ferrate and ferroso-ferric oxide at a resonably high temperature (about 1300°C). Thus it is quite expected that in glass system the thermal treatment affects the magnetic property of the glass. The magnetic data of some such glasses along with their thermal history are given in the following table.

It is thus seen from Table VIII that the susceptibility value of iron in glass increases with the increase of temperature of melting and the duration of heating and that the increase of susceptibility value of iron is due to the increase in and formation of some forms of iron with high mass susceptibility as well as the simultaneous growth of colloidal dispersoid like Fe_2O_3 if there be any, in the vitreous matrix. In this context the absence of ferromagnetic solid phase colours is shown by making the susceptibility measurements of the above samples at different field strengths of 3500 gauss and 2500 gauss respectively

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